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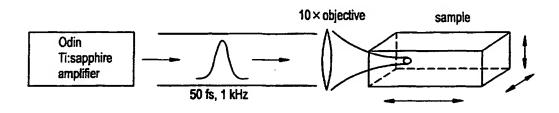
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(54) Title: DIRECT WRITING OF OPTICAL DEVICES IN SILICA-BASED GLASS USING FEMTOSECOND PULSE LASERS





(57) Abstract: The invention relates to methods of writing a light guiding structure in a bulk glass substrate. The bulk glass substrate is preferably made from a soft silica-based material having an annealing point less than about 1380 °K. Pulsed laser beam is focused within the substrate while the focus is translated relative to the substrate along a scan path at a scan speed effective to induce an increase in the refractive index of the material along the scan path. Substantially no laser induced physical damage of the material is incurred along the scan path. Various optical devices can be made using this method.

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# DIRECT WRITING OF OPTICAL DEVICES IN SILICA-BASED GLASS USING FEMTOSECOND PULSE LASERS

#### CROSS REFERENCE TO RELATED APPLICATIONS

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This application claims priority to the United States Provisional Patent Application Serial No. 60/146,274, filed July 29, 1999, entitled Direct Writing of Optical Devices In Silica-Based Glass Using Femtosecond Pulse Lasers of Nicholas F. Borrelli and Charlene Smith and the United States Provisional Patent Application Serial No. 60/172,122, filed December 17, 1999, Femtosecond Laser Writing Of Glass, Including Borosilicate, Sulfide and Lead Glasses of Nicholas F. Borrelli, David L. Morse, Alexander Streltsov and Bruce Aitken.

#### **BACKGROUND OF THE INVENTION**

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The present invention relates to methods for efficiently forming optical devices in glass. Specifically, the invention relates to direct-write methods of forming light guiding structures in glass compositions through light-induced refractive index changes using pulsed lasers having a pulse duration less than about 150 femtoseconds. The invention also relates to the optical devices made by the direct-write methods.

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Optical devices such as optical waveguides and Bragg diffraction gratings are widely known in the telecommunications field. In an optical waveguide, a higher refractive index core surrounded by a lower refractive index cladding can transmit a large amount of optical information over long distances with little signal attenuation. The optical waveguide fiber is the prototype device of this type. The fiber is produced

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by a method that, by virtue of its fabrication, gives the proper waveguiding structure. A Bragg grating is another type of optical device that can be used to isolate a narrow band of wavelengths from a broader signal. The most common materials used commercially in telecommunications applications of light guiding devices are doped silica-based compositions.

It is known that pulsed laser sources can be used to effect both index changes and to produce physical damage in glass. With regard to the former, the use of pulsed UV radiation sources for writing Bragg gratings is known. Recently, a "direct-write" laser method of forming optical waveguides within a glass volume that is transparent to the wavelength of a femtosecond laser has been disclosed. In this method, a 120 fs pulsed 810-nm laser is focused within a polished piece of germania-doped silica as the glass is translated perpendicular to the incident beam through the focus. Increases in refractive index on the order of 10<sup>-2</sup> were reported for a specific condition in which the focus was scanned ten times over the exposed area.

One potential problem with a direct write process of forming waveguides in bulk glass using short-pulse focused lasers is over-exposure. Irradiation with too much energy can lead to physical damage in the glass. Physical damage results in undesired attenuation of optical signals transmitted through the glass.

Another problem in direct write methods of making optical structures relates to the trade-off between the dimensional stability of the writing device, e.g., the laser, and the energy necessary to induce the desired refractive index change in the substrate material. Femtosecond lasers can be operated in three modes. Each of these has advantages and disadvantages associated with it. The properties of each laser configuration also make materials more or less desirable for a certain application. The table below presents some of the characteristics of these different modes.

|                       | Amplifier  | Oscillator | Cavity Dump                        |
|-----------------------|------------|------------|------------------------------------|
| Pulse Duration (fsec) | 40 – 150   | <40        | <40                                |
| Energy Range          | 1 μJ – 1mJ | 1 – 10 nJ  | 10 – 50 nJ                         |
| Rep Rate              | >kHz       | >MHz       | <1MHz                              |
| Mode Quality          | Poor       | Good       | Good                               |
| Stability             | Poor       | Good       | Not as stable as oscillator system |

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The table above illustrates the operational trade-offs as a consequence of how the laser is configured. While it is relatively easy to obtain a 100 MHz repetition rate using the oscillator mode when the pulse energy is less than 10 nJ, at the  $\mu$ J level of energy the repetition rate is traded off and drops to the several kHz range. Mode quality, which is qualitatively described by the temporal and spatial integrity of the beam, is relatively poor in the amplified system and improves when the oscillator is used. Similarly, the overall stability of the laser is found to be more robust in the oscillator case. These parameters turn out to be of practical importance in direct-write methods of making optical devices where one needs to control the dimensional stability of the laser beam in order to write closely spaced optical structures in the substrate, such as diffraction grating lines.

To make the femtosecond laser direct-write method practical, large changes in the refractive index (>10<sup>-3</sup>) of a material must be achieved in a reasonable amount of writing time. The formation of laser-induced physical damage should also be avoided. There continues to be a need for a practical direct write method of creating silicabased optical devices having a sufficiently increased refractive index at an acceptably high write rate. Such a method could be used to write continuous light-guiding waveguide patterns connecting any two points within a continuous block of a suitable material, or make other optical devices, such as Bragg gratings.

#### BRIEF SUMMARY OF THE INVENTION

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It is an object of the invention to provide improved direct-write methods of forming light guiding structures within a silica-based material substrate. In particular, it is desired to efficiently write three-dimensional light guiding structures in glass, such a waveguides and gratings, more quickly using lower pulse energy levels without incurring physical damage of the glass.

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It is a further object of the invention to write optical structures in silica-based materials using dimensionally stable operating modes of ultra-fast lasers.

In accordance with one aspect of the invention, it has been discovered that soft silica-based materials exhibit increased sensitivity to ultra-fast laser writing of optical

structures in the bulk. In particular, femtosecond laser-induced refractive index changes can be more easily produced in silica-based compositions having an annealing point that is lower than that of the 5 mol.% germania (GeO<sub>2</sub>) - 95 mol.% silica (SiO<sub>2</sub>) system in that lower pulse energies and faster translation speeds can produce equivalent increases in refractive index as harder silica-based materials.

In accordance with another aspect of the invention, a method is provided to directly write light guiding structures in glass using short-pulse lasers with substantially no physical damage of the glass.

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In accordance with another aspect of the invention, a method is provided to write three dimensional optical structures in silica-based bulk glass. Specifically, the invention provides for translating the refractive index-increasing focus of an ultra-fast laser through a silica-based substrate in the x, y, and z-dimensions.

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In accordance with still another aspect of the invention, a variety of optical devices are disclosed which incorporate optical structures made by the methods described herein.

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These and other aspects of the invention will become apparent to those skilled in the art in light of this disclosure.

#### BRIEF DESCRIPTION OF THE SEVERAL VIEWS OF THE DRAWINGS

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- Fig. 1 is a schematic arrangement of equipment used in practicing the invention.
- Fig. 2(a) and Fig. 2(b) show the positioning of the incident laser beam relative to the scan direction in the top-write and axial-write orientations, respectively.
- Fig. 3(a) and Fig. 3(b) show the scanning beam profile and a photograph of waveguides cross-sectional shape in the top-write orientations, respectively.

- Fig. 3(c) and Fig. 3(d) show the scanning beam profile and a photograph of waveguides cross-sectional shape in the axial-write orientations, respectively.
- Fig. 4(a) and Fig. 4(b) are perspective views of the top-write arrangement of directly writing three dimensional optical devices in bulk glass.

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Fig. 5 is a schematic drawing of the equipment set-up for observing the far-field pattern.

Fig. 6 is a photograph of a far-field intensity pattern of a waveguide written in a silica-based material according to the invention.

Fig. 7 is a photograph of a far-field intensity pattern of a waveguide written in borate-doped silica according to the invention.

Figs. 8(a) - 8(b) are photographs of near-field intensity patterns of waveguides written in fused silica, germania-doped silica.

Fig. 8(c) is a trace of the intensity of the near field pattern and borate-doped silica.

Figs. 9(a) - 9(d) show various exemplary optical devices that can be made using the invention.

Fig. 10 is a photograph of a Y-coupler written in silica using the invention.

#### 15 DETAILED DESCRIPTION OF THE INVENTION

The direct-write method of forming light guiding structures in a bulk substrate according to the invention includes the steps of selecting a substrate made from a silica-based material in which the light guiding structure is to the written, focusing a pulsed laser beam at a position within the substrate effective to induce an increase in the refractive index of a portion of the irradiated material, and translating the substrate and focus with respect to one another to form a light guiding structure within the substrate along the scan path.

The method may be better understood by reference to a generalized arrangement of an equipment set-up suitable for practicing the invention, as shown in Fig. 1. Laser 1 generates a pulsed laser beam 2 which is focused at a focus 3 positioned within a glass sample 4 by a lens 5. The sample is translated in one or more of the x-direction 6, y-direction 7, and z-direction 8 to effect translation of the sample with respect to the laser beam focus at a desired translation or scan speed. Such translation of the sample with respect to the focal point may be accomplished by a positioning or translation device (not shown), such as a computer controlled XYZ stage.

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Focusing of the laser beam significantly increases the peak intensity of the beam compared to an unfocused beam. The high intensity of the focused beam effects an increase in the refractive index of the glass along the path traced by the beam focus as it is translated through the sample. The resulting region of increased refractive index can guide light and therefore can function as an optical waveguide.

A "top writing" method results from translating the sample in a scan direction 13 that is substantially perpendicular to the incident beam, as shown in Fig. 2(a). An "axial writing" method results from translating the sample in a scan direction 13 that is substantially parallel to the incident beam, as shown in Fig. 2(b). As the skilled artisan will readily appreciate, top-writing may also be accomplished by translating the sample in just the x-direction, just the y-direction, or both the x-direction and y-direction simultaneously.

The focus profile and cross-sectional shape of top-written waveguides shown in Fig. 3 (a) and Fig. 3(b) differ from those of axial-written waveguides, as shown in Fig. 3(c) and Fig. 3(d). The beam profile in the vicinity of the focus relative to the scan direction 13 is shown for the top-write orientation in Fig. 3(a) and for the axial-write orientation in Fig. 3(c), respectively. When the top-write focus is translated through the sample in the scan direction, a generally elliptoid cross-section of the waveguide may be produced, as indicated by Fig. 3(b). When the axial-write focus is translated through the sample in the scan direction, a generally circular cross-section of the waveguide often results, as indicated by Fig. 3(d). Accordingly, axially-written waveguides are generally preferred in order to produce waveguides having substantially circular cross-sections. Top-writing may be desired in order to write continuous linear waveguides longer than the focal length of the focusing lens.

The ability to write three-dimensional waveguides in a sample using the present direct-write method is described further with reference to Figs. 4(a) and 4(b). The laser beam 2 can be focused by a lens 5 to a focus 3 positioned within glass sample 4. Translation of the sample in the x-, y-, and z-directions from a first position  $(x_1,y_1,z_1)$  at depth  $D_1$  to a second position  $(x_2,y_2,z_2)$  at depth  $D_2$  causes an increase in the refractive index of the glass along the scan path 9 to form an optical waveguide extending in three dimensions between the first and second positions within the sample. If planar, i.e., two-dimensional, waveguides are desired,  $x_1$  may be the same

as  $x_2$ ,  $y_1$  may be the same as  $y_2$ , or  $z_1$  may be the same as  $z_2$ . If linear waveguides are desired,  $x_1$  and  $y_1$  may be the same as  $x_2$  and  $y_2$  respectively,  $y_1$  and  $z_1$  may be the same as  $y_2$  and  $z_2$ , respectively, or  $x_1$  and  $z_1$  may be the same as  $x_2$  and  $z_2$ , respectively.

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The pulsed laser beam is characterized by several beam parameters. The beam parameters include the wavelength, pulse duration or pulse width, pulse energy, and repetition rate. Preferably, the laser wavelength and sample are selected to minimize optical absorption of the beam energy by the sample. In the case of both doped and undoped silica-based glasses, the wavelength can fall within the range of about 400 nm to about 1100 nm, preferably from about 800 nm to about 830 nm. Within this range of wavelengths, the optical absorption of the beam by a silica-based sample is virtually nonexistent. The glass materials intended to be used with this invention are substantially transparent to the wavelengths of interest.

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The time duration of each pulse, a.k.a., the pulse width, is preferably less than about 150 fs. Lasers having pulse widths of this duration or shorter are referred to as femtosecond or ultra-fast lasers. More preferably, the pulse duration is less than about 100 fs. Most preferably, the pulse width is about 40 fs to about 60 fs. Lasers having pulse widths as short as 18 fs have been used to practice the invention. The energy per pulse, or pulse energy, can be from about 1 nJ to about 10  $\mu$ J, preferably within the range of about 0.1 to about 10  $\mu$ J. More preferably, the pulse energy falls within the range of about 1 to about 4  $\mu$ J. The repetition rate or pulse frequency preferably falls within the range extending from about 1 kHz to about 250 kHz for amplified laser systems, but can be as high as 80 MHz.

The laser can be any device capable of generating a pulsed laser beam characterized by the desired beam parameters. The laser may be, for example, a Ti:Sapphire amplifier system. One suitable laser is a Quantronix Odin multipass amplifier seeded with a mode-locked Ti:sapphire oscillator.

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A suitable focusing lens includes a microscope objective having a magnification power of about 5x to about 20x. The focusing lens preferably has a numerical aperture (NA) of about 0.16 to about 0.25. An especially preferred focusing lens is a

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10x, 0.16 NA aspheric lens. A diffraction limited spot size of the focused laser beam was achieved using this lens.

The translation device may be any device capable of translating the sample with respect to the beam focus at the translation speeds of interest. Preferably, the translation speed lies in the range of about 5  $\mu$ m/s to about 500  $\mu$ m/s or faster. For example, a computer controlled XYZ positioning device, available from the Newport Co., can be used.

While the examples below refer to moving the glass sample with respect to a fixed focus, the skilled artisan will readily appreciate that alternatively the laser focus could be moved relative to a fixed sample, or both the laser focus and sample could be moved simultaneously with respect to a fixed reference point to achieve the desired relative translation speed between the sample and focus.

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While the drawings have depicted the glass samples suitable for use in the present invention as having substantially planar surfaces oriented at right angles to one another, the skilled artisan will recognize that the invention is not limited to such regular solid geometries. Rather, the invention can be used to direct-write optical waveguides in virtually any regular- or irregular-shaped three-dimensional sample. It is preferred, however, that the sample be positioned relative to the incident laser beam such that the beam is substantially perpendicular to the surface of the sample through which the incident beam passes.

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The composition of the substrates in which the light guiding structures may be written by this invention are silica-based materials, including undoped fused silica and doped binary and ternary silica systems. Silica-based materials are preferred in light of their various desirable optical properties as well as their widespread use in telecommunication device applications.

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By "silica-based materials" we mean glass compositions that include silica and which are essentially free of alkali, alkaline earth, and transition metal elements, as well as other impurities which would cause absorption in the 1300 – 1600 nm range. If present at all, such impurities will typically not be found in the silica-based materials used in this invention at levels higher than 10 ppb (parts per billion).

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In accordance with this disclosure, waveguides can be written more easily in bulk substrates made from soft silica-based glass compositions using lower pulse energies and/or faster translation speeds than in hard silica-based materials without sacrificing the magnitude of the induced index change. Soft silica-based compositions appear to be more sensitive to direct writing of light guiding structures using ultra-fast (femtosecond) lasers than hard silica-based composition glasses.

For the purposes of this disclosure, "soft" silica-based materials are defined as doped or undoped silica-based materials having an annealing point less than that of 5 mol.% GeO<sub>2</sub> – 95 mol.% SiO<sub>2</sub>, i.e., silica-based materials having an annealing point less than about 1380°K. The preferred silica-based glasses are undoped and doped binary or ternary silica-based materials having an annealing point less than about 1380°K, more preferably less than about 1350°K, and most preferably within the range of about 900°K to about 1325°K. The annealing point is defined as the temperature at which the viscosity of the material is 10<sup>13.8</sup> poise.

Undoped soft silica-based materials include, for example, commercial grade fused silica, such as Corning 7980 glass, which can have an annealing point in the range of about 1261°K to about 1323°K. As for the doped systems, the preferred dopants which may be used to soften silica include oxides of the elements boron, phosphorous, aluminum, and germanium, such as borate (B<sub>2</sub>O<sub>3</sub>), phosphate (P<sub>2</sub>O<sub>5</sub>), alumina (Al<sub>2</sub>O<sub>3</sub>), and germania (GeO<sub>2</sub>), respectively.

In binary boron-doped silica-based systems, the borate content may comprise up to 20 wt.% or more borate. For example, the binary glass systems 9 wt.%  $B_2O_3$ -91 wt.%  $SiO_2$  and 20 wt.%  $B_2O_3$ -80 wt.%  $SiO_2$  may be used to practice the invention. The annealing point of the 9 wt.%  $B_2O_3$ -91 wt.%  $SiO_2$  composition is about 1073°K. The annealing point of the 20 wt.%  $B_2O_3$ -80 wt.%  $SiO_2$  composition is about 999°K.

In binary phosphorous-doped silica-based systems, the phosphate content may also comprise up to 20 wt.% or more phosphate. For example, the binary glass systems 10 wt.%  $P_2O_5$ -90 wt.%  $SiO_2$  and 7 wt.%  $P_2O_5$ -93 wt.%  $SiO_2$  may be used to

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practice the invention. The annealing point of the 7 wt.%  $P_2O_5$ -93 wt.%  $SiO_2$  composition is about 1231°K.

In binary aluminum-doped silica-based systems, the alumina content may comprise up to 20 wt.% or more alumina. For example, the binary glass systems 10 wt.% Al<sub>2</sub>O<sub>3</sub>-90 wt.% SiO<sub>2</sub> may be used to practice the invention.

In binary germanium-doped silica-based systems, the germania content may comprise up to about 22 wt.% or more germania. For example, the binary glass systems 20 wt.% GeO<sub>2</sub>-80 wt.% SiO<sub>2</sub> and 22 wt.% GeO<sub>2</sub>-78 wt.% SiO<sub>2</sub> may be used to practice the invention. The annealing point of the 20 wt.% GeO<sub>2</sub>-80 wt.% SiO<sub>2</sub> composition is about 1323°K while that of the 22 wt.% GeO<sub>2</sub>-78 wt.% SiO<sub>2</sub> composition is about 1311°K.

"Hard" silica-based materials are defined as doped or undoped silica-based materials having an annealing point higher than that of the 5 mol.% GeO<sub>2</sub> – 95 mol.% SiO<sub>2</sub> system, i.e., higher than about 1380°K. Examples of hard silica-based materials include dry fused silica which has an annealing point of about 1425°K. As is generally known in the art, "dry" fused silica has virtually no residual hydroxyl groups, while commercial grade fused silica may have higher levels, for example, about 800 ppm hydroxyl groups.

The skilled artisan will readily appreciate that many other silica-based compositions could be used to practice the invention.

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The silica-based materials used in this invention are preferably made by a flame hydrolysis process. In such a process, silicon-containing gas molecules are reacted in a flame to form SiO<sub>2</sub> soot particles. These particles are deposited on the hot surface of a rotating body where they consolidate into a very viscous fluid which is later cooled to the glassy (solid) state. In the art, glass making procedures of this type are known as vapor phase hydrolysis/oxidation processes or simply as flame hydrolysis processes.

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The induced refractive index changes reported below in connection with the examples were determined by the beam spread method assuming a step index profile. A schematic of the experimental set-up for estimating the radiation-induced change in the refractive index in the waveguides made according to the invention by this method is shown in Fig. 5. After writing a waveguide 16 in sample 4, by using a spatial filter 20, collimating lens 19, beam splitter 17, telescope 18, and lens 5, light from a HeNe laser 21 was coupled into the waveguide 16 and the numerical aperture (NA) of the cone of light that emerged was measured. Since the length of the waveguides made in the example below was typically 1 cm, unguided light from the HeNe interfered with the light coupled out the waveguide. This interference resulted in an interference pattern of concentric rings in the far field as recorded by a digital camera 14 and personal computer 15. A recorded image of the interference pattern is shown in Fig. 6.

The radius at which the fringes died out, R<sub>fringe</sub>, was measured. The distance from the exit of the waveguides to the viewing surface, L, was fixed at 75 cm. The NA of the waveguide was calculated from the relation

Assuming a step index profile, the induced refractive index change  $\Delta n$  was then calculated based on the relation  $\Delta n = (NA)^2 / 2n$ .

In order that the invention may be more readily understood, reference is made to the following examples, which are intended to be illustrative of the invention, but are not intended to be limiting in scope.

#### Example 1

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Pulses from a Ti:sapphire multi-pass amplifier which were 60-fs in duration and had pulse energies of approximately 1 μJ were focused with a 10x (0.16 NA) microscope objective into fused-silica glass samples mounted on a computer controlled high-precision 3-D translation stage. The fused-silica samples were translated through the focal point of the beam at a rate of 30 μm/s. Waveguide structures were created within the bulk material.

A 830 nm laser was used to deliver 40 fs pulses at a 1 kHz repetition rate. The energy per pulse was from about 1  $\mu$ J to about 5  $\mu$ J. The beam was focused into the glass below the surface with a lens with a numerical aperture of 0.16 in air. The sample was moved under the beam at a rate of about 5 µm/s to about 100 µm/s. The experimental conditions were kept constant for exposure to samples of fused silica and for 14 wt.% GeO<sub>2</sub> – 86 wt.% SiO<sub>2</sub>. The beam was focused about 1 mm below the surface of the glass. For samples irradiated at the same exposure conditions, the diameter of the laser-affected region of the germania-silica sample was about twice that of the fused silica sample. From this result, we concluded that the germania-silica material was more sensitive to refractive index changes induced by ultra-fast laser exposure than fused silica.

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#### Example 3

Substrates of various glass compositions, i.e., SiO<sub>2</sub> (Corning product 7980), 22 wt.% GeO<sub>2</sub>-78 wt.% SiO<sub>2</sub> and 9 wt.% B<sub>2</sub>O<sub>3</sub>-91 wt.% SiO<sub>2</sub>, were exposed to focused laser radiation by the axial write method. The laser wavelength was 830 nm. The pulse duration was 40 fs. The energy per pulse was 1.0 uJ. The repetition rate was 1 kHz. The scan speed was 20 μm/s. After exposure, the induced refractive index change at 633 nm was estimated from the far-field pattern of the waveguide produced. The induced refractive index change results are tabulated below in Table 1. The annealing point of each of these materials is also reported in Table 1.

Table 1: Induced refraction index change (Example 3)

| Glass Composition                                    | Annealing Point | Induced Refractive |
|--|-----------------|--------------------|
| (% based on weight)                                  | (°K)            | Index Change       |
| SiO <sub>2</sub> (Corning 7980)                      | 1261 - 1323     | 0.0003             |
| 78%SiO <sub>2</sub> -22%GeO <sub>2</sub>             | 1311            | 0.0009             |
| 91%SiO <sub>2</sub> -9%B <sub>2</sub> O <sub>3</sub> | 1073            | 0.0030             |

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#### Example 4

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A sample of 9 wt.%  $B_2O_3$ -91 wt.%  $SiO_2$  glass was exposed to focused laser radiation by the axial write method. The laser wavelength was 830 nm. The pulse duration was 40 fs. The energy per pulse was 1.0  $\mu$ J. The repetition rate was 1kHz. The scan speed was 20  $\mu$ m/s. A photomicrograph of the far field pattern of this sample is shown at Fig. 7. The double lobed pattern is indicative of the propagation of a higher order mode. Insofar as the silica and germania samples of Example 2 showed single lobe patterns, the effective refractive index change of the borate sample must have been greater than that of the other two samples to support the additional mode.

#### Example 5

Each of the glass compositions listed in Table 1 were exposed to focused laser radiation. The laser wavelength was 830 nm. The pulse duration was 40 femtoseconds. The energy per pulse was 0.5 μJ. The scan speed was 10 μm/s. After exposure, the samples were photographed through a microscope at a magnification of 400x. The resulting photomicrographs of the SiO<sub>2</sub>, 22 wt.% GeO<sub>2</sub> – 78 wt.% SiO<sub>2</sub>, and 9 wt.% B<sub>2</sub>O<sub>3</sub> – 91 wt.% SiO<sub>2</sub> samples at Fig. 8(a) to 8(c), respectively, show increasing spot sizes for the softer glass compositions. These results indicate the increased sensitivity of the softer glass compositions to 40 fs pulsed laser irradiation at 830 nm.

The foregoing results strongly suggest that softness of the exposed glass compositions is a key parameter in determining the magnitude of the laser induced refractive index change.

#### Example 6

Optical waveguides were written in various bulk glasses using femtosecond laser irradiation. A Ti-sapphire laser irradiating at 830 nm with a pulse width within the range of about 40 fs to about 50 operating at pulse energies of 0.5 - 10 μJ. The pulse repetition rate was 1 kHz. The beam was focused with a 0.15 NA lens into the block of glass that was translated at linear speeds of 5 - 100 μm/s. Assuming a diffraction

limited Gaussian beam, the estimated spot size of the focal point of the beam was 5  $\mu$ m. The glass was exposed to the beam by translating the block relative to the focal point in the axial direction, i.e., in the direction of the beam. The nominal intensities used for the exposure therefore ranged from 0.05 - 1 x 10<sup>15</sup> W/cm<sup>2</sup>. The induced refractive index changes (10<sup>-3</sup>) are reported in Table 2.

Table 2: Induced refractive index change (10<sup>-3</sup>) (Example 6)

| Glass<br>(% based on weight)              | Pulse Scan Speed (μm/s) t) |      |         |      | m/s) |     |
|---|----------------------------|------|---------|------|------|-----|
|   | (Lu)                       | 100  | 50      | 20   | 10   | 5   |
|   | 2                          | 0.4  | -       | -    | -    | -   |
| SiO <sub>2</sub>                          | 1                          | 0.2  | 1.2     | 2.4  | 0.8  | -   |
|   | 0.5                        | -    | 0.03    | 0.2  | 0.4  | 0.6 |
|   | 2                          |      | 0.5     | 2.6  |      |     |
| 9%GeO₂ - 91%SiO₂                          | 1                          | ·    | -       | 1    | 0.5  |     |
|   | 0.5                        |      |         |      |      |     |
|   | 2                          | 0.16 | 0.9     | 6    | 16   | -   |
| 22%GeO <sub>2</sub> - 78%SiO <sub>2</sub> | 1                          | 0.01 | 0.2-0.5 | 0.9  | 4-10 | ~   |
|   | 0.5                        | -    | -       | 0.5  | 1-2  | 5   |
|   | 2                          | 1    | 4*      | 8    |      |     |
| 9%B₂O₃ -91%SiO₂                           | 1                          | 1    | 3-4     | -10* | 10*  |     |
|   | 0.5                        |      |         | 0.04 | 0.1  |     |
|   | 2                          | -    | -       | -    | -    | -   |
| SiO₂ (hydrogen loaded)                    | 1                          | •    | -       | 0.3  | 1.3  | -   |
|   | 0.5                        | -    | -       | -    | -    | -   |

<sup>\*</sup> double lobed pattern

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Optical waveguides were written in fused silica using femtosecond laser irradiation. The Ti-sapphire laser irradiated at 830 nm with a 150 fs pulse width. The pulse energy was 5, 10, and 20  $\mu$ J. The pulse repetition rate was 1 kHz. The beam was focused with a 0.1 NA lens. The glass substrate was translated at linear speeds of 15, 50, and 500  $\mu$ m/s. The glass was exposed to the beam by translating the substrate relative to the focus in the "top-write" orientation, i.e., in a direction perpendicular to the beam. The induced refractive index changes (10<sup>-3</sup>) for Example 7 are reported in Table 3.

Table 3: Induced refractive index change (10<sup>-3</sup>) (Example 7)

| Glass | Pulse Energy | Scan Speed (μm/s) |     |    |
|-------|--------------|-------------------|-----|----|
|       | (لىر)        | -500              | 50  | 15 |
|       | 20           | 0.1               |     |    |
| SiO₂  | 10           |                   | 0.9 | 3  |
|       | 5            |                   | 0.4 |    |

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Femtosecond laser pulses were produced by a Quantronix Odin multipass amplifier which was seeded with a mode-locked Ti:sapphire oscillator. The operating wavelength was 830 nm. The system produced 60-fs pulses at a 1 kHz repetition rate. The laser beam was focused into a sample of fused silica using a 10x (0.16 NA) single aspheric-lens microscope objective. Photonic structures were written by translating the sample with respect to the focal region using computer controlled three-dimensional stages which had a resolution of 200 nm. By using this objective having this relatively long working-distance, waveguides as long as 2 cm parallel to the beam were written. Using the NA measurement techniques described above, values for the induced refractive index change ( $10^{-3}$ ) of the silica were determined, as reported in Table 4. In all cases, the diameter of the waveguides was approximately 3  $\mu$ m. The waveguide diameter appeared to have minimal dependence on the incident pulse energy or the translation speed.

Table 4: Induced refractive index change (10<sup>-3</sup>) in fused silica (Example 8)

| Pulse Energy | Scan Speed (μm/s) |      |      |     |       |      |      |
|--------------|-------------------|------|------|-----|-------|------|------|
| (ئىر)        | 400               | 200  | 100  | 50  | 20    | 10   | 5    |
| 4.0          | •                 | -    | 0.83 |     | 3.3*  |      |      |
| 2.0          | •                 | -    | 0.03 | 0.3 | 2.1*  | 2.5* |      |
| 1.0          | •                 | -    | -    | -   | 0.065 | 1.1  | 0.97 |
| 0.5          | -                 | 1 1- | -    |     | -     | -    | 0.53 |

<sup>\*</sup> double-lobed far-field intensity pattern that is characteristic of a second mode

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The experimental conditions of Example 8 were repeated, but the sample was made of the softer glass composition 9 wt.%  $B_2O_3 - 91$  wt.%  $SiO_2$  rather than fused silica. The values for the induced refractive index change (10<sup>-3</sup>) of the boron-doped silica material are reported in Table 5.

Table 5: Induced refractive index change (10<sup>-3</sup>) in boron-doped silica (Example 9)

| Pulse Energy | Scan Speed (μm/s) |     |       |       |       |      |             |
|--------------|-------------------|-----|-------|-------|-------|------|-------------|
| (لىر)        | 400               | 200 | 100   | 50    | 20    | 10   | 5           |
| 4.0          | 1.2               | 1.7 | 4.03* |       |       |      | <del></del> |
| 2.0          | 1.1               | 1.4 | 2.5   | 4.03* | 3.3*  | 4.8* |             |
| 1.0          | -                 | 1.1 | 1.4   | 3.3*  | 4.83* | 3.3* | T           |
| 0.5          | -                 | -   | 0.13  | 0.83  | 2.1*  |      |             |

<sup>\*</sup> double-lobed far-field intensity pattern that is characteristic of a second mode

In most cases, the same write conditions, including pulse energy and scan speed produced a larger induced refractive index increase in the boron-doped silica glass than in the fused silica glass. Accordingly, the exposure required to produce the same degree of index change is significantly less for the boron-doped silica material then for the fused-silica material.

<sup>-</sup> too small to measure

<sup>-</sup> too small to measure

The increased sensitivity of the boron-doped glass compared to the fused silica glass is illustrated also by comparing the exposure required to produce the characteristic double-lobed far-field pattern as shown in Fig. 6. This pattern appears to correspond to the onset of a second mode.

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It is interesting to note that the onset of the second mode for a simple step-index waveguide is given by the equation  $2\pi rNA/\lambda = 2.4$ . We measure that at the onset of the double-lobed pattern the NA is 0.08, and with the measuring wavelength of 633 nm this value would correspond to a waveguide radius of about 3  $\mu$ m, which is approximately the size of the observed guide. In both the boron-doped silica and the pure fused silica glasses, the response of the material appears to saturate, and attempts to produce index changes larger than the saturation value by either increasing the pulse energy or by reducing the translation speed resulted in damaged waveguides that did not efficiently guide light.

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A wide variety of optical devices in bulk glass can be made using the presently described materials and methods. Example 10 describes the fabrication and performance of a Y-coupler device.

#### 20 Example 10

A Y-coupler was written in a bulk sample of pure fused silica at the conditions of Example 1. A photograph of the structure shows the guiding of light from an argon laser, as shown in Fig. 10. The vertical dimension of the photograph is magnified with respect to the horizontal dimension for clarity. The splitting angle was measured as approximately 0.5°. It was observed that approximately half of the 514.5 nm light was coupled into each of the two branches of the coupler.

The present invention can also be used to make a wide variety of other optical devices, such as the star coupler having central guide 22 surrounded by a plurality of peripheral guides 23, as shown in Fig. 9 (a). The invention can also be used to make a passive Mach-Zehnder coupler including a pair of Mach-Zehnder guides 26, as shown in Fig. 9(b). An active Mach-Zehnder coupler including Mach-Zehnder guides 26 and a thermal or other type activator 24, as shown in Fig. 9(c), could also be made using this invention.

The present invention can also be used to make Bragg or the type diffraction gratings in bulk glass, as shown in Fig. 10. Waveguide 16 leads to grating lines 25. Line spacings of  $0.5~\mu m$  are possible using this invention.

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It will be understood that the above described preferred embodiment(s) of the present invention are susceptible to various modifications, changes, and adaptations, and the same are intended to be comprehended within the meaning and range of equivalents of the appended claims.

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Further, although a number of equivalent components may have been mentioned herein which could be used in place of the components illustrated and described with reference to the preferred embodiment(s), this is not meant to be an exhaustive treatment of all the possible equivalents, nor to limit the invention defined by the claims to any particular equivalent or combination thereof. A person skilled in the art would realize that there may be other equivalent components presently known, or to be developed, which could be used within the spirit and scope of the invention defined by the claims.

#### CLAIMS:

- 1. A method of writing a light guiding structure in a bulk glass substrate comprising:
  - a) selecting a bulk glass substrate made from a soft silica-based material; and
- b) focusing a pulsed laser beam at a focus within said substrate while translating the focus relative to the substrate along a scan path at a scan speed effective to induce an increase in the refractive index of the material along the scan path relative to that of the unexposed material while incurring substantially no laser induced breakdown of the material along the scan path.
- 10 2. The method of claim 1 wherein said material has an annealing point lower than about 1350°K.
  - 3. The method of claim 2 wherein the material has an annealing point lower than about 1325°K.
- 4. The method of claim 2 wherein the material is substantially transparent to the laserwavelength.
  - 5. The method of claim 2 wherein the ratio of the band gap of the material to the energy of the laser irradiation is at least about 5.
  - 6. The method of claim 2 wherein the peak intensity of said laser beam at the focus is at least about 10<sup>14</sup> W/cm<sup>2</sup>.
- 7. The method of claim 2 wherein the material includes a first dopant selected from the group consisting of GeO<sub>2</sub>, B<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub> and P<sub>2</sub>O<sub>5</sub>.
  - 8. The method of claim 7 wherein said material further includes a second dopant different in composition from said first dopant, said second dopant being selected from the group consisting of GeO<sub>2</sub>, B<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, and P<sub>2</sub>O<sub>5</sub>.
- 25 9. The method of claim 2 wherein the laser pulse duration is from about 18 fs to less than 120 fs.
  - 10. The method of claim 2 wherein the laser repetition rate is from about 1 kHz to less than 200 kHz.
  - 11. The method of claim 2 wherein the pulse energy is within the range from about 1 nJ to about 10 μJ.
  - 12. The method of claim 11 wherein the pulse energy is within the range from about 1 μJ to about 4 μJ.
  - 13. The method of claim 11 wherein the pulse energy is within the range from about 1 nJ to about 10 nJ.

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- 14. The method of claim 2 wherein the scan speed is greater than 20  $\mu$ m/s and less than about 500  $\mu$ m/s.
- 15. The method of claim 2 wherein the focus is translated relative to the substrate in a scan direction that is substantially parallel to the laser beam.
- 5 16. The method of claim 2 wherein the focus is translated relative to the substrate in a scan direction that is substantially perpendicular to the laser beam.
  - 17. The method of claim 2 wherein the focus is translated relative to the substrate in three dimensions.
  - 18. The method of claim 2 wherein the diameter of the light guiding structure is about 3 μm to about 4 μm.
  - 19. The method of claim 2 wherein translation of the focus once along the scan path induces a refractive index increase of more than about 0.0001.
  - 20. A product made by the process of claim 2.
- 21. The product of claim 20 wherein the product is a device selected from the group consisting of a Y-coupler, a directional coupler, a star coupler, a Mach-Zehnder device, a loop mirror, a demux coupler, a Er-doped single- or multi-stage amplifier, and devices having surface-modified thermal, piezoelectric, or trench-type activators.
  - 22. A diffraction grating made by the process of claim 2.
- 20 23. The product of claim 22 wherein the line spacing is about 0.5  $\mu$ m.
  - 24. A method of writing a light guiding structure in a bulk glass substrate comprising:
    - a) selecting a bulk glass substrate made from a hard doped silica-based material;
       and
    - b) focusing a pulsed laser beam at a focus within said substrate while translating the focus relative to the substrate along a scan path at a scan speed effective to induce an increase in the refractive index of the material along the scan path relative to that of the unexposed material while incurring substantially no laser induced breakdown of the material along the scan path.
  - 25. The method of claim 24 wherein the material is substantially transparent to the laser wavelength.
  - 26. The method of claim 24 wherein the ratio of the band gap of the material to the energy of the laser irradiation is at least about 5.
  - 27. The method of claim 24 wherein the peak intensity of said laser beam at the focus is at least about 10<sup>14</sup> W/cm<sup>2</sup>.

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- 28. The method of claim 24 wherein the material includes GeO<sub>2</sub>.
- 29. The method of claim 24 wherein the laser pulse duration is from about 18 fs to less than 120 fs.
- 30. The method of claim 24 wherein the laser repetition rate is from about 1 kHz to less than 200 kHz.
- 31. The method of claim 24 wherein the pulse energy is within the range from about 1 nJ to about 10 μJ.
- 32. The method of claim 31 wherein the pulse energy is within the range from about 1 ய to about 4 ய.
- 10 33. The method of claim 31 wherein the pulse energy is within the range from about 1 nJ to about 10 nJ.
  - 34. The method of claim 24 wherein the scan speed is greater than 20  $\mu m/s$  and less than about 500  $\mu m/s$ .
- 35. The method of claim 24 wherein the focus is translated relative to the substrate in a scan direction that is substantially parallel to the laser beam.
  - 36. The method of claim 24 wherein the focus is translated relative to the substrate in a scan direction that is substantially perpendicular to the laser beam.
  - 37. The method of claim 24 wherein the focus is translated relative to the substrate in three dimensions.
- 20 38. The method of claim 24 wherein the diameter of the light guiding structure is about 3 μm to about 4 μm.
  - 39. The method of claim 24 wherein translation of the focus once along the scan path induces a refractive index increase of more than about 0.0001.
  - 40. A product made by the process of claim 24.
- 25 41. A method of writing a light guiding structure comprising:
  - a) selecting a bulk glass substrate including a silica-based material made by a flame hydrolysis process;
  - b) focusing a pulsed laser beam at a focus within said substrate while translating the focus relative to the substrate along a scan path at a scan speed effective to induce an increase in the refractive index of the material along the scan path relative to that of the unexposed material while incurring substantially no laser induced breakdown of the material along the scan path.
  - 42. A method of writing a light guiding structure in a bulk glass substrate comprising:

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- a) selecting a bulk glass substrate made from a silica-based material doped with a dopant selected from the group consisting of B<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub> and P<sub>2</sub>O<sub>5</sub>; and
- b) focusing a pulsed laser beam at a focus within said substrate while translating the focus relative to the substrate along a scan path at a scan speed effective to induce an increase in the refractive index of the material along the scan path relative to that of the unexposed material while incurring substantially no laser induced breakdown of the material along the scan path.
- 10 43. A method of making a three dimensional internal tunnel light guiding structure within an interior of a glass body, said method comprising:

providing a glass body, said glass body having an interior, said interior having a homogeneous composition and refractive index,

providing a pulsed laser beam,

focusing said pulsed laser beam to form a converging focused laser beam having a refractive index increasing focus,

positioning said focus inside said glass body interior and controlling relative motion between said focus and said glass body, wherein said focus forms a raised refractive index waveguiding core structure which tunnels through said glass body, said raised refractive index waveguiding core for guiding light and cladded by said glass body.

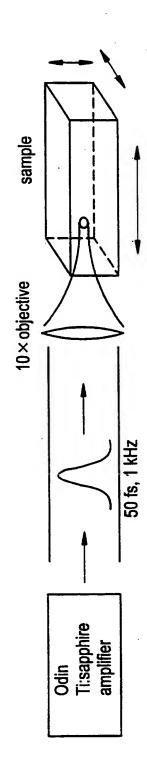
- 44. A method as claimed in claim 43, said glass body having a first exterior side and a second exterior side, said first exterior side lying in a first plane, said second exterior side lying in a second plane, said second plane non-parallel to said first plane, wherein said waveguiding core tunnels from an input at said first exterior side to an output at said second exterior side.
- 45. A method as claimed in claim 43, said glass body having a planar exterior base side, wherein said waveguiding core tunnels in a plane non-parallel to said planar base side.
  - 46. A method as claimed in claim 43, said method including forming a first raised refractive index waveguiding core tunnel path, a second raised refractive index waveguiding core tunnel path, and a third raised refractive index waveguiding core

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tunnel path, wherein said third tunnel path is in a plane separate from said first tunnel path and said second tunnel path.

- 47. A method as claimed in claim 43, said providing a glass body including providing a glass homogeneously doped with a glass softening dopant.
  - 48. A method as claimed in claim 43, wherein said focus forms a refractive index increase of at least 1 X 10<sup>-5</sup>.
- 10 49. A method as claimed in claim 43, wherein said focus forms a refractive index increase of at least 1 X 10<sup>-4</sup>.
- 50. A method as claimed in claim 43, said method including forming a first raised refractive index waveguiding core tunnel path and a second raised refractive index
   15 waveguiding core tunnel path wherein guided light is coupled from said first core tunnel path to said second core tunnel path.
- 51. A method as claimed in claim 43, wherein said method includes forming a wavelength division multiplexer for multiplexing a plurality of optical wavelength
  20 channels, said forming including forming a plurality of waveguiding core tunnel inputs for separately inputting the plurality of optical wavelength channels, forming a multiplexing region for multiplexing said inputted channels, and forming an output waveguiding core tunnel for outputting said multiplexed inputted channels.

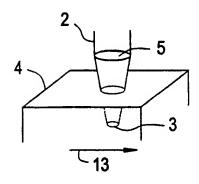
FIG.1



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FIG.2A





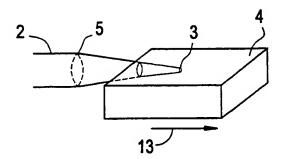
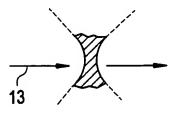


FIG.3A

FIG.3C



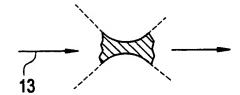


FIG. 3B

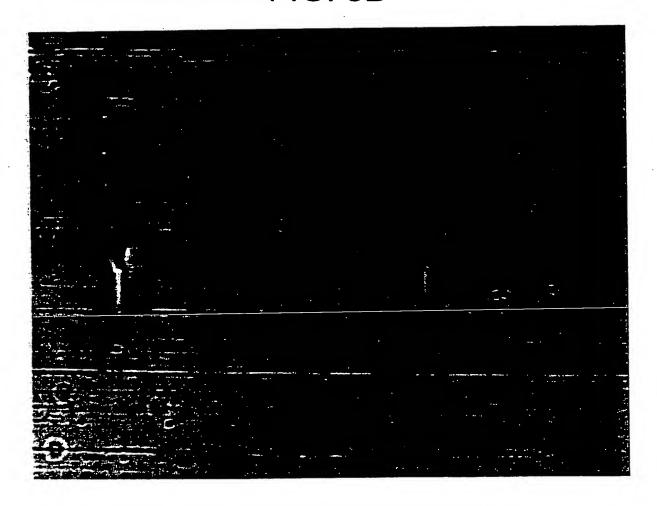


FIG. 3D

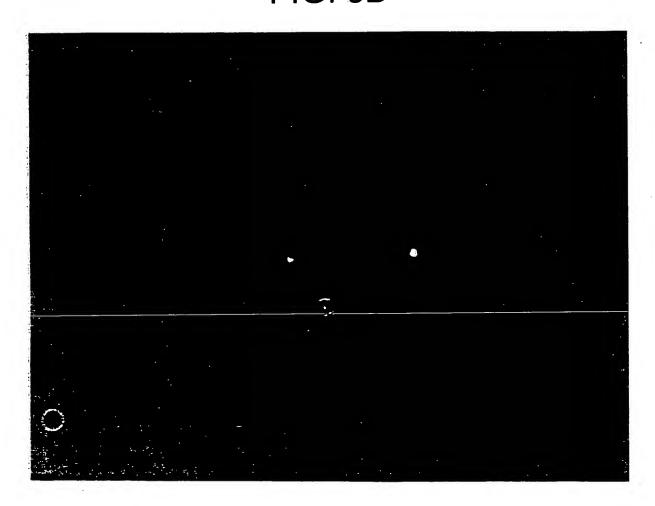


FIG.4A

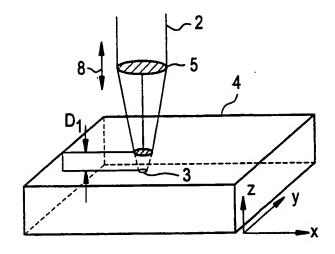


FIG.4B

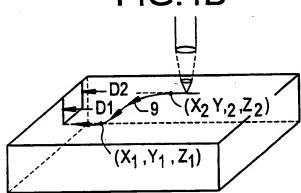


FIG.5

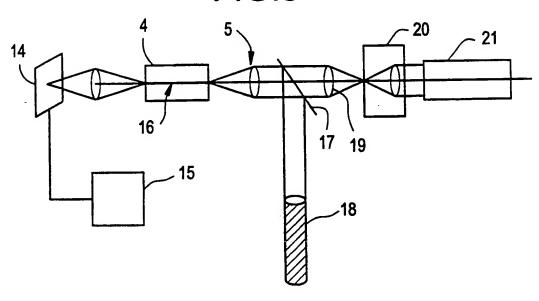


FIG. 6

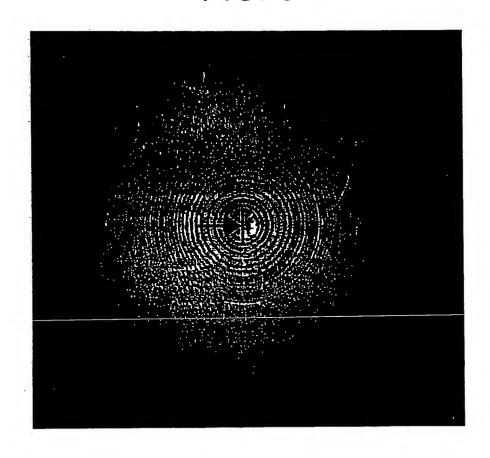


FIG. 7

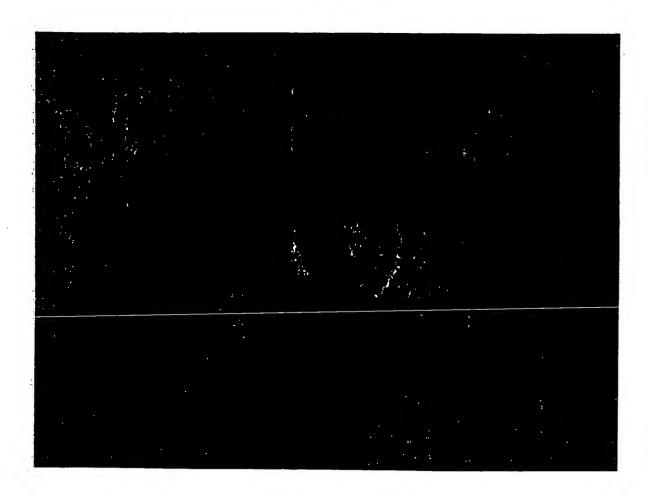


FIG. 8A

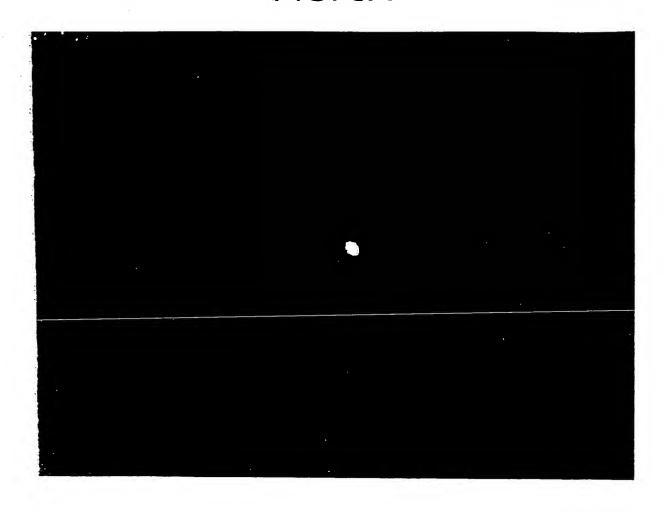
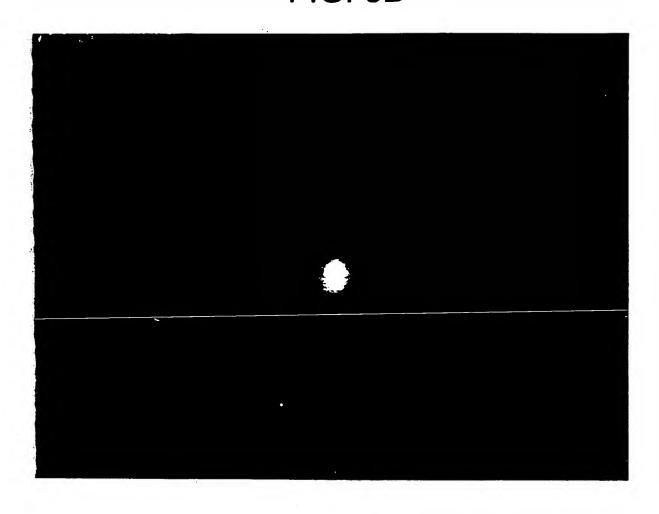
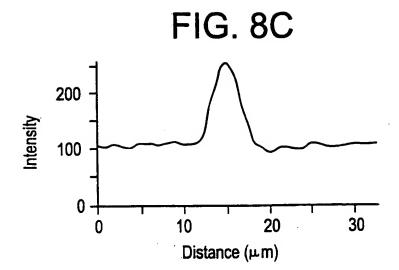
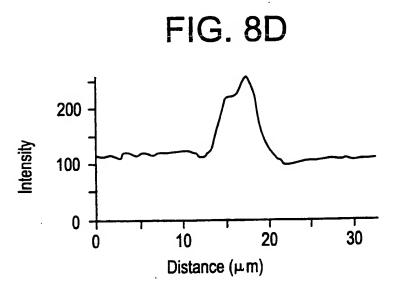


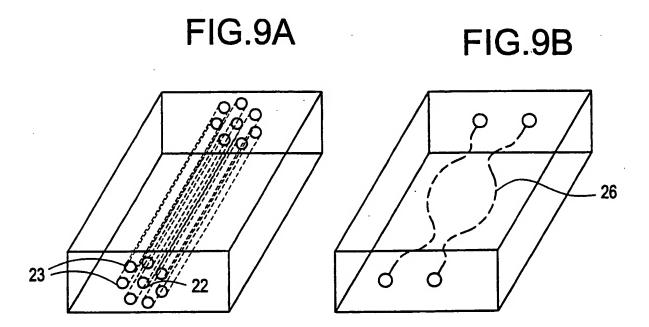
FIG. 8B



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24 V 26 26

FIG. 9C

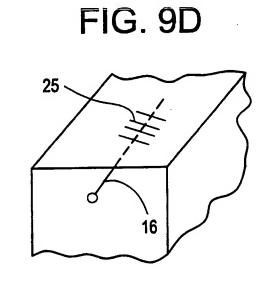
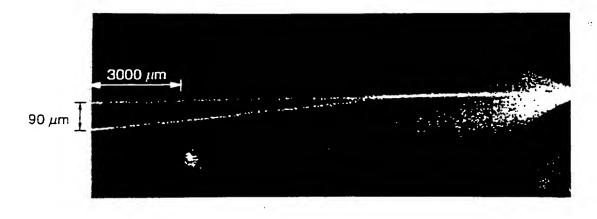


FIG. 10



#### INTERNATIONAL SEARCH REPORT

International application No. PCT/US00/20446

| A. CLASSIFICATION OF SUBJECT MATTER  IPC(7) :G11C 13/04  US CL :219/121.6,121.85; 365/106,127  According to International Patent Classification (IPC) or to both national classification and IPC |  |   |                  |  |  |  |  |  |
|--|--|---|------------------|--|--|--|--|--|
|  | B. FIELDS SEARCHED  Minimum documentation searched (classification system followed by classification symbols)  |   |                  |  |  |  |  |  |
|  | 219/121.6.121.85,121.67,121,68,121,69; 365/106,127   |   | /A0              | ;  |  |  |  |  |
| U.S. :   | 219/121.6,121.83,121.67,121,68,121,09, 303/100,127   | ,123, 204/462, 34/1223, 3/2   | 740              |  |  |  |  |  |
|  | ion searched other than minimum documentation to the   |   |                  |  |  |  |  |  |
| Electronic d   | lata base consulted during the international search (na  | me of data base and, where  | practicable.     | search terms used)   |  |  |  |  |
| C. DOC   | UMENTS CONSIDERED TO BE RELEVANT   |   |                  |  |  |  |  |  |
| Category*  | Citation of document, with indication, where ap  | propriate, of the relevant pas  | ssages           | Relevant to claim No.  |  |  |  |  |
| A  | US 5,157,674 A (LAWANDY) 20 Oc   |   | 1-51             |  |  |  |  |  |
| A  | US 5,253,198 A (BIRGE et al) 12 Oct  |   | 1-51             |  |  |  |  |  |
| A  | US 5,289,407 A (STRICKLER et al)   | 1-51  |                  |  |  |  |  |  |
| A  | US 5,325,324 A (RENTZEPIS et al) 2   | 1-51  |                  |  |  |  |  |  |
| A  | US 5,761,111 A (GLEZER) 02 June 1  | 998   |                  |  |  |  |  |  |
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| Furth  | er documents are listed in the continuation of Box C   | . See patent fami   | ly annex.        |  |  |  |  |  |
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|  | "P" document published prior to the international filing date but later than the priority date claimed "&" document member of the same patent family |   |                  |  |  |  |  |  |
| Date of the  | Date of the actual completion of the international search  Date of mailing of the international search report  |   |                  |  |  |  |  |  |
| 12 OCTO  | 12 OCTOBER 2000 02 NOV 2000  |   |                  |  |  |  |  |  |
| Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT  Authorized officer SAMIEL M. HEINBICH   |  |   |                  |  |  |  |  |  |
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